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Kwasny, J., McGrath, T., Lawther, S., Soutsos, M., Sha, W., Cox, S., Chen, J. F., & Correia Lopes, R. F. (2019). Lagoon fly ash: A potential source of the precursor for geopolymer binders. In M. J. McCarthy, M. D. Newlands, M. R. Jones, T. D. Dyer, L. J. Csetenyi, & L. Zheng (Eds.), *EUROCOALASH2019 Proceedings* (pp. 322-333).

Published in:
EUROCOALASH2019 Proceedings

Document Version:
Peer reviewed version

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LAGOON FLY ASH – A POTENTIAL SOURCE OF THE PRECURSOR FOR GEOPOLYMER BINDERS

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ABSTRACT: The use of lagoon fly ash (KLFA) from the Kilroot power station in Northern Ireland was investigated as a potential source of precursor in alkali activated geopolymer concretes. Chemical composition, particle size distribution, mineralogy and morphology of the received KLFA sample were evaluated and compared to that of the silo-stored fly ashes obtained from Kilroot (KFA) and Drax (DFA) power stations. Mortar mixes were proportioned with these ashes and tested for compressive strength to assess their reactivity in the geopolymerisation process. Specifically, mixes made with KLFA samples in as received state (wet), oven-dried state and processed states (dried and milled) were compared with mixes proportioned with silo-stored fly ashes. Subsequently, samples to test the variability of the fly ash in the Kilroot lagoon were collected from different locations during a geological survey and then their chemical compositions were determined.

Samples of KLFA in the as received state and KFA had similar chemical composition, mineralogy and particles size distribution. However, large clumps of fly ash particles within KLFA were found. The strength of the mix made with KLFA in the processed state was comparable to that of the silo fly ash mixes, whereas KLFA used in the as received state and oven-dried state was deemed unsuitable. Most KLFA samples collected during the survey showed lower content of silicon and/or higher loss on ignition value than KFA, which could potentially adversely affect geopolymerisation. Thus, KLFA is suitable to use as a raw material for alkali activation, however preselection and processing prior to use is required.

Keywords: lagoon fly ash, chemical and physical characterisation, geopolymer, precursor, reactivity.

Authors:

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INTRODUCTION

Geopolymer binders, being a sub-group of alkali activated binders (AABs), are a novel class of construction materials. They offer an environmentally friendly alternative to Portland cement-based binders and have been reported to have better/equivalent mechanical and durability properties when compared to Portland cement materials [1]. AABs, including geopolymers, are produced by reacting an alumino-silicate rich precursor with an alkali-silicate solution, called a chemical activator [1]. For geopolymers, an inorganic polymerisation reaction results in the formation of an amorphous aluminosilicate hardened matrix. Typically, the geopolymer precursor is a waste or an industrial by-product, the most common being a neat fly ash, a blend of fly ash/slag or a calcined clay [1-3].

Fly ash is a fine powder extracted from flue gas by electrostatic precipitators or bag filters during the combustion process in coal-fired power stations. It is then either stored in silos for use in different engineering materials and projects or is unused, *i.e.* it is landfilled. The UK supply of good quality fly ash for concrete applications is limited [4]. Over 4.6 Mt of fly ash was produced in the UK in 2014, of which *ca.* 50% was used in production of Portland cement or concrete products, 30% was landfilled and remaining 20% was used in other applications (mostly geotechnical) [5]. The quantity of fly ash has most likely dwindled, and will continue to do so, due to the governmental strategy to move away from fossil fuels for electricity generation [6]. For example, reduction in coal demand from 38 Mt in 2014 to 12 Mt in 2016 has already been seen [7]. Therefore, a continuous demand of fly ash for use in blended cements, or as partial replacement of Portland cement, will cause increased pressure on its supplies [4]. Heath *et al.* [8] anticipated that current global production of fly ash and slag meets only 20% of Portland cement demand and will most likely fall below 10% by 2050. Therefore, a reliable source of fly ash has to be secured, particularly to allow for an uptake of geopolymer technology.

It is estimated that UK lagoons and landfills may contain >100 Mt of fly ash [9]. Literature indicates that prolonged storage of the fly ash in a lagoon/landfill can cause significant chemical changes (alteration to composition due to weathering and leaching of easy dissolvable salts) and physical changes (segregation, creation of agglomerates and deposition of reaction products on the surface of fly ash particles), adversely affecting its pozzolanic reactivity [9-11]. Loss on ignition (LOI) values of most lagoon/landfilled stored ashes are high and prevent their use in Portland cement applications in the UK [9]. Therefore, a number of studies were undertaken to evaluate a revalorisation of the UK lagoon/landfilled stored fly ashes in Portland cement systems, highlighting an additional processing (drying/screening) of the material that is likely to be required [9, 11]. However, there is limited work available on the suitability of using lagoon/landfilled ashes in geopolymer systems.

The objective of the work presented in this paper was (1) to characterise the fly ash that has been stored in the lagoon of the Kilroot Power Station and compare its properties with silo-stored ashes, and (2) to assess the potential of using this ash as a source of precursor for geopolymer binders.

EXPERIMENTAL PROGRAMME

Materials and Methods

The work was divided into two phases.

In the first phase, the activation potential of the Kilroot lagoon fly ash (KLFA) as precursor in geopolymer binders was assessed. The chemical composition, particle size distribution, mineralogy and morphology of KLFA were evaluated. Then, geopolymer mortar mixes were prepared to test compressive strength at 1-, 7- and 28-days (Table 1). Three mortar mixes were made with KLFA in as-received moist state (KLFA-W), oven-dried state (KLFA-D), and with a sample of KLFA which was oven-dried and then milled (KLFA-DM). Performance of the mortar made with KLFA was compared to mortars proportioned with silo-stored fly ashes obtained from Kilroot (KFA) and Drax (DFA) power stations.

Geopolymer mixes were proportioned with a liquid chemical activator comprising a water solution of sodium hydroxide and a sodium silicate solution. The sodium hydroxide solution was produced by dissolving laboratory grade pellets of sodium hydroxide (NaOH) in water (30% by mass). A commercially available sodium silicate solution with the following mass percentage composition was used: 15% Na₂O, 30% SiO₂ and 55% water.

Table 1 Fly ash-based geopolymer mixes used to determine activation potential of KLFA

MIX CODE	ASH SOURCE	APPLIED TREATMENT
GDFA	DFA	Used as received (dry)
GKFA	KFA	Used as received (dry)
GKLFA-W	KLFA-W	Used as received (wet) and sieved on 2 mm sieve
GKLFA-D	KLFA-D	Oven-dried [#] and sieved on 2 mm sieve
GKLFA-DM	KLFA-DM	Oven-dried [#] and milled ^{##}

[#] – Dried at 105 °C to remove excess moisture

^{##} – Milled in a Retsch PM400 Ball Mill at 300 rpm for 10 minutes

Well-graded 0–5 mm concrete sand, with oven-dry particle density of 2695 kg/m³, was sourced locally in Northern Ireland (Creagh Concrete Products Ltd., Draperstown). Water absorption of sand at 1-hour and 24-hours was 0.92% and 1.1%, respectively. Both density and water absorption were determined according to BS 812-2:1995 [12].

In phase two, further 12 samples of KLFA were collected during a pilot geological survey at Kilroot site to assess the variability in their chemical composition.

Mix Composition

All geopolymer mortar mixes were made with a chemical activator having the same composition: alkali dosage (M+) and alkali modulus (AM). The value of M+, *i.e.* the percentage mass ratio of total sodium oxide (Na₂O) in the chemical activator to the precursor, was fixed at 7.5%. The value of AM, *i.e.* the mass ratio of sodium oxide to silica in the chemical activator, was set at 1.25.

The water to solids (w/s) ratio of the geopolymer mortars was defined as the ratio between mass of the mixing water (*i.e.* mass of water in the chemical activator + mass of added water) and mass of the solids (*i.e.* mass of the precursor + mass of solids in the chemical activator). This ratio was kept constant at 0.35. The sand to precursor mass ratio was fixed at 2.75. Sand was pre-saturated before mixing with extra water (quantity equal to the 1-hour water absorption of sand; this extra water was not included in w/s ratio calculations), to achieve its saturated-surface-dry state.

Mix Preparation, Sample Casting, Demoulding and Conditioning

To ensure that no other parameters influenced the results, all constituent materials were stored at room temperature (20 ± 2 °C) prior to batching. Before mixing, sand was oven-dried (at 105 ± 5 °C) until a constant mass was reached, subsequently cooled and stored in sealed plastic barrels.

The mortar mixes were made in a Hobart mixer in 1 L batches. Pre-saturated sand (based on its 1-hour water absorption) and precursor were placed in the mixing bowl and mechanically

homogenised for 1 minute. The designed quantities of the sodium hydroxide solution, sodium silicate solution and added water were mixed together and then added to the mixing bowl. The geopolymer mortar was mixed for a total of 6 minutes.

All mortar specimens were cast in two layers. Each layer was compacted on a vibrating table. After casting, the moulds with samples were wrapped in polythene plastic sheets and placed in the oven at 70 °C. Samples were demoulded at 24 ± 0.5 hours, counting from the casting time, and placed in plastic boxes on 15 mm plastic supports. Boxes were filled with water to the height of 5 mm, then covered with tightly fitting lids and stored in the conditioning room (20 ± 1 °C). This procedure allowed the conditioning of the samples at relative humidity of >95% and prevented unintentional carbonation of the samples, and leaching of alkalis.

Test Techniques

Particle size distribution analysis of fly ashes was carried out using a Malvern Mastersizer in the 0–2000 µm range. Before testing all samples were oven-dried and sieved on 1 mm sieve.

Samples for XRF and XRD spectroscopy analyses of ashes were transferred to a desiccator and stored for *ca.* 24 hours under vacuum at 40 ± 1 °C to evaporate the moisture. Then, dried samples of KLFAs were powdered using mortar and pestle to pass a 63 µm sieve. Immediately after grinding, the powdered samples were placed in sealable plastic bags and stored in the desiccator under vacuum at 20 ± 1 °C until testing.

X-ray fluorescence (XRF) spectroscopy was used to determine the chemical composition of the ashes. Samples were analysed at the University of Leicester using PANalytical Axios Advanced XRF spectrometer.

Powdered samples were analysed using powder XRD spectroscopy, with a PANalytical X'Pert PRO diffractometer, to identify the crystalline components of ashes. Diffraction patterns were collected between 2θ of 5 and 65° with a step size of 0.016°. PANalytical X'Pert Highscore software with the Powder Diffraction File database was used to identify the mineralogy of the samples based on the diffraction patterns.

Morphology of fly ashes was assessed by scanning electron microscopy (SEM) with energy dispersive X-ray (EDX) analysis. A QUANTA FEG250 scanning electron microscope with OXFORD X-Act chemical composition analyser was used.

Compressive strength of mortar cubes at given ages (1-day, 7-day and 28-day) was determined by crushing two 50×50×50 mm cubes each time (at a constant loading rate of 200 kN/min). The average of two measurements is reported in MPa.

RESULTS AND DISCUSSION

Activation Potential of Lagoon Fly Ash

Appearance and moisture content

The received sample of KLFA had clumps of ash particles throughout the material (size varied, up to several mm in diameter – Figure 1a). This occurred as a result of the wet

storage in the lagoon. These clumps typically broke down when pressed between fingers. As expected, the moisture content of the sample was very high, exceeding 21%. Therefore, the KLFA will potentially require additional processing before being used as a raw material in alkali activated binders. In comparison, the silo-stored ashes (KFA and DFA) were in dry, powder form and could be used with no additional processing required.

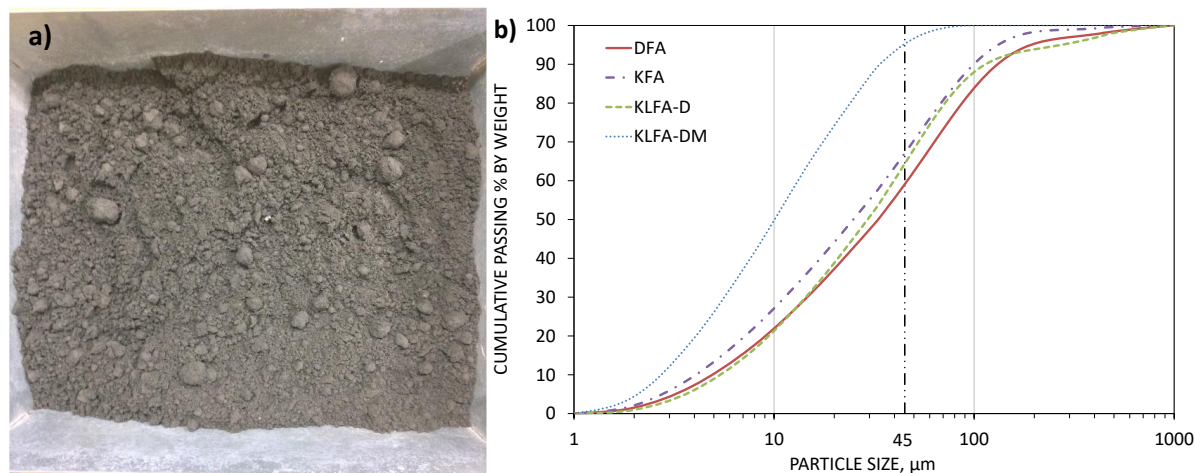


Figure 1 a) received sample of KLFA with visible large clumps of ash particles, b) particle size distribution of the investigated lagoon- and silo-stored ashes

Chemical composition and loss on ignition

Chemical compositions and loss on ignition (LOI) values of three different ashes used in this phase of work (KLFA, KFA and DFA) are given in Table 2. To compare the chemical composition of the silo-stored KFA, Table 2 also shows composition of two extra KFA samples (KFA* and KFA**) sourced at different dates. The major oxides of these fly ashes, identified using XRF spectroscopy, were as follows: SiO₂, Al₂O₃, Fe₂O₃ and CaO. SiO₂ of all samples varied between 46.78 and 56.32%, with ashes obtained from Kilroot Power station having SiO₂ above 50%. The Al₂O₃ of all ashes varied in a relatively narrow range: from 22.06 to 23.56%. DFA had the highest Fe₂O₃ content of 9.15%, whereas Kilroot ashes had it between 4.72 and 6.9%.

Table 2 Chemical composition and LOI of ashes tested during KLFA activation potential

COMPOUND [%]	KLFA	KFA	KFA*	KFA**	DFA
SiO ₂	52.06	50.54	56.32	55.38	46.78
Al ₂ O ₃	22.22	22.06	23.54	23.56	22.52
Fe ₂ O ₃	5.84	6.90	4.72	4.76	9.15
CaO	3.97	5.54	4.39	4.69	2.24
MgO	1.81	2.03	1.8	1.79	1.33
MnO	0.07	0.09	0.05	0.05	0.05
TiO ₂	0.96	0.82	1.04	1.03	1.05
Na ₂ O	0.76	1.06	1.01	0.88	0.89
K ₂ O	2.02	1.79	1.81	1.86	4.09
P ₂ O ₅	0.55	0.77	0.73	0.71	0.17
SO ₃	0.50	<0.002	0.56	0.55	0.9
LOI	7.88	7.194	3.58	3.74	3.57

The SiO₂ content and sum of SiO₂, Al₂O₃ and Fe₂O₃ for each of the ashes was $\geq 25\%$ and $\geq 70\%$ respectively. The total CaO content was $\leq 10\%$, whereas the total content of alkalis ($\text{Na}_2\text{O}_{\text{eq}} = \text{Na}_2\text{O} + 0.658 \cdot \text{K}_2\text{O}$, all in mass %) was $\leq 5\%$. The contents of other minor oxides were as follows: MgO $\leq 4\%$, P₂O₅ $\leq 5\%$ and SO₃ $\leq 3\%$. Therefore, all ashes provisionally comply with BS EN 450-1 [13] the general chemical composition requirements (contents of Cl⁻ and reactive CaO to be confirmed).

Loss on ignition (LOI) values of KLFA and KFA were fairly high ($>7\%$) when compared to that of DFA and the other two KFA samples, *i.e.* KFA* and KFA** ($<3.8\%$). When LOI limits of BS EN 450-1 [13] are concerned, samples of KLFA and KFA would fall in category C (LOI $\leq 9\%$), whereas DFA and the two other samples of KFA (KFA* and KFA**) would fall in category A (LOI $\leq 5\%$). Currently, BS EN 8500-2 [14] allows LOI $\leq 7\%$ (category A and B) for use of fly ashes in Portland cement concrete.

Particle size distribution

The particle size distributions of raw KLFA (KLFA-D, dried and sieved on 1 mm sieve) and processed KLFA (KLFA-DM, dried and milled) are shown in Figure 1b. They are compared to grading of two silo-stored ashes, *i.e.* KFA and DFA. The particle size distributions of KLFA-D, KFA and DFA were fairly similar, although KLFA-D had lower proportion of particles passing 10 μm and higher content of particles passing 105 μm . Overall, KLFA-DM had the finest particle size distributions with all particles passing 105 μm , whereas KFA was finer than DFA.

Based on the BS EN 450-1 [13] requirements, KLFA-D, KFA and DFA can be classified into the fineness category N ($\leq 40\%$ mass of particles $>45 \mu\text{m}$) and KLFA-DM into the category S ($\leq 12\%$ mass of particles $>45 \mu\text{m}$). In general, reactivity of fly ashes is related to particle size. Therefore, the above results can aid the interpretation of the compressive strength data.

Mineralogy

Results of the XRD analysis of KLFA, KFA and DFA samples are shown in Figure 3. All three samples had very similar XRD traces. The compositions of the materials were very similar and all contained the characteristic ‘amorphous hump’ centred at 2θ of approximately 25° . DFA shows traces of hematite that are not present in KLFA and KFA. Calcite (CaCO₃) was present in KLFA. Given that calcite was not found in KFA, it is likely that it was formed within KLFA due to prolonged exposure to the atmospheric CO₂ and weathering [15].

Morphology

SEM imaging was carried out on the ash samples of KLFA, KFA and DFA (Figure 2). All ashes were very similar in appearance, regardless of the source, primarily containing spherical fly ash particles. The spheres varied in size, from several to tens of microns, and some of the larger spheres were coated with smaller spheres. The surface of the spheres was typically smooth, but some spherical particles had pitted surface. Spot analysis of the spherical particles showed that they were primarily composed of Si and Al (in different particles the proportion of Si to Al varied, but Si was generally dominant). Within the KLFA the spheres often appear as clumps of particles (varying largely in their number) with no apparent material holding them together. This potentially may affect the reactivity of KLFA.

reduction of the grain size is well known to increase the reactivity of fly ash [2, 16]. However, these results indicate that after mechanical activation KLFA was not as reactive as KFA. In summary, KLFA could be suitable to use as a raw material for the alkali activation, with prior processing.

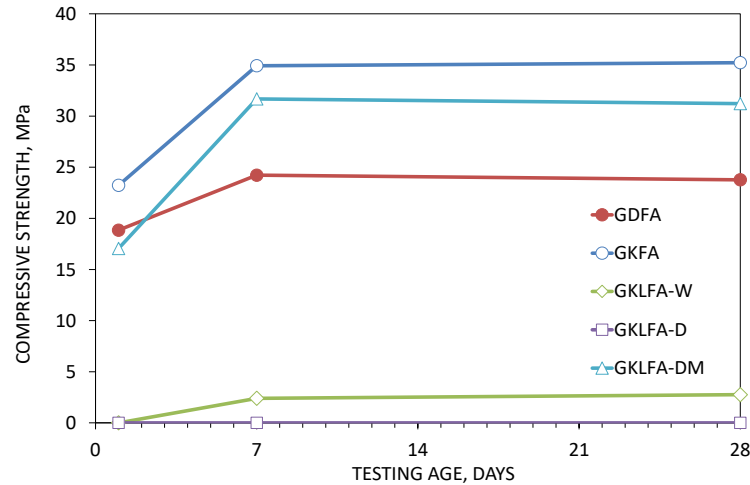


Figure 4 Compressive strength development of fly ash geopolymer mortars

Variability of Lagoon Fly Ash at Kilroot Power Station

Sampling of Kilroot lagoon fly ashes

A preliminary auger sampling program was organised to test the variability of KLFA. Samples were obtained from 12 different sites within the lagoon (Figure 5). The auger was capable of sampling to the depth of 2 m, hence the initial plan was to obtain samples from different height levels within the lagoon. This was considered useful, as if there would be variability across the site within the top 2 m, then it is likely that the variability would continue to the depth.



Figure 5 Aerial photograph of Kilroot lagoon showing approximate sampling locations

When sampling commenced it was clear that the ground was extremely compacted and that sampling would be problematic. Using the auger a maximum depth of 50 cm was achieved and at some sites sampling was from the top couple of centimetres as the ash could not be penetrated with the equipment at hand (Figure 5). For two sites samples were obtained using an excavator, which enabled sampling from the depth of approximately 2.5 m.

Chemical composition of Kilroot lagoon fly ashes

XRF analysis was carried out on 12 samples collected from the Kilroot lagoon. Selected XRF results are shown in Figure 6 (separated into two graphs for the ease of reading) and are compared to the ash samples listed in Table 2. The horizontal lines represent averages calculated for each elemental oxide/LOI for the silo-stored samples of KFA, KFA* and KFA** (Table 2), enabling easy comparison of Kilroot silo- and lagoon-stored ashes.

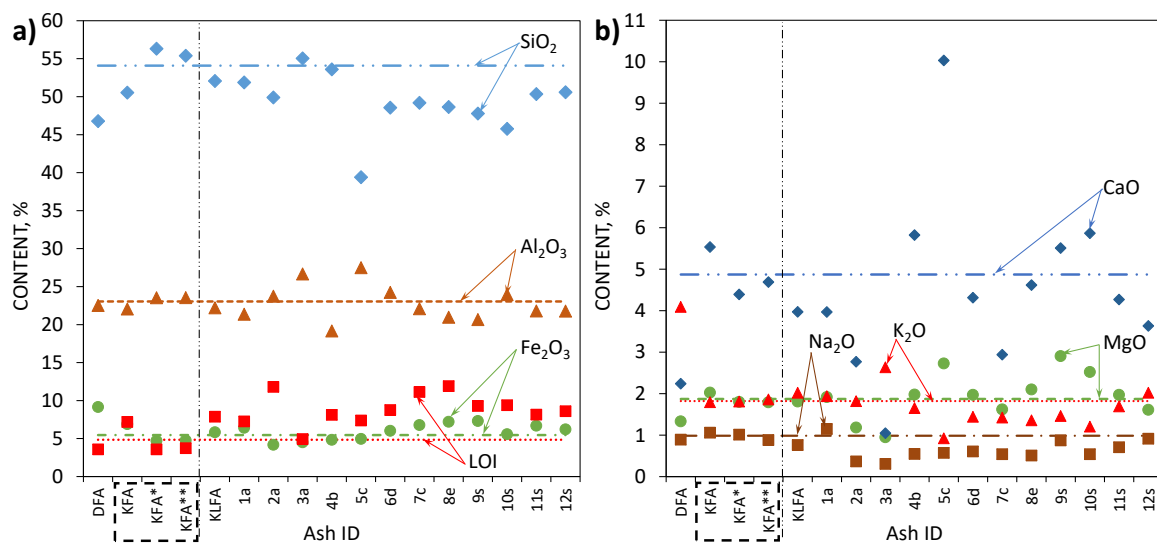


Figure 6 XRF of selected oxides and LOI in ashes shown in Table 2 and 12 KLFA samples

Results of the greatest importance for geopolymerisation are those for silica, aluminium and LOI (Figure 6a). In general, SiO₂ content of KLFA samples was below the KFA average (ranging from 39.40 to 55.04%), whereas Al₂O₃ content was close to the KFA average (ranging from 22.06 to 24.75%). LOI was increased compared to that observed in silo KFAs (ranging from 4.95 to 11.91%). As discussed previously, increased LOI in KLFA samples can be related to the amount of unburnt carbon and to weathering-related carbonation.

Other oxides that show significant changes were Fe₂O₃ (Figure 6a), CaO, MgO, Na₂O, K₂O (Figure 6b), TiO₂, P₂O₅, SO₃ (not depicted on graphs). CaO, Fe₂O₃ was variable throughout KLFA samples. MgO and TiO₂ were typically above average and Na₂O, K₂O, P₂O₅ and SO₃ below the average. Kilroot lagoon is located by the sea and thus fluctuations in MgO, TiO₂, Na₂O, K₂O, P₂O₅ and SO₃ are possible, depending on the extent of interaction with seawater. Samples 5c and 10s in the KLFA show a significant increase in CaO, MgO, P₂O₅ and SO₃, which points towards the seawater interacting with these samples. In these samples the SiO₂ content was also decreased more than in any of the other samples. This may indicate that interactions with seawater result in decreased SiO₂ content.

Given that geopolymerisation requires high levels of SiO₂ within the system, there is a possibility that seawater interaction reduces the SiO₂ content and may result in the ash being unsuitable for geopolymerisation. Therefore, to ascertain this, it is necessary to carry out trial mixes with ash collected from different sites/depths within the lagoon and assess the effect of the seawater on the strength development of these mixes, and on the geopolymerisation process in general. Further study of lagoon fly ash would also require the chloride levels to be tested, as chlorides can be detrimental for durability of reinforced concrete.

CONCLUSIONS

Based on the results, the following conclusions have been reached:

- As received KLFA sample had similar chemical composition, mineralogy and particle size distribution to silo-stored KFA. SEM imaging revealed large clumps of fly ash particles within KLFA, which lead to low reactivity of KLFA when used in geopolymer mortars in the unprocessed form. Drying and milling of KLFA increased its reactivity and allowed it to achieve a similar strength to the geopolymer mix made with KFA.
- The variation in the chemical composition of the KLFA samples collected during the survey was noticeable, however it depended on the specific oxide. Regarding the geopolymer binders, the content of SiO₂ in KLFAs was lower than in KFAs, whereas Al₂O₃ content was similar to that of KFAs. LOI values of KLFAs were increased compared to that observed in KFAs. Effect of seawater on the KLFA properties and geopolymerisation process has to be assessed across the lagoon (different sites/depths).
- KLFA is suitable to use as a raw material for some types of alkali activation, however preselection and processing of KLFA prior to use is required.

Acknowledgements

The work reported here is part of the ongoing research project (LowCoPreCon – Low carbon footprint precast concrete products for an energy efficient built environment) at Queen's University Belfast funded by the Newton-Ungku Omar Fund which is supported by Innovate UK and Malaysian Industry-Government Group for High Technology (MIGHT). The authors are grateful to the School of Natural and Built Environment for the facilities provided and to Innovate UK for the financial support.

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